

polarity value⁴ - 124 to "standard" SP2250); and (ii) 3% polyethylene glycol 20M on Supelcoport 80-100 mesh in glass 1.5 m × 4 mm I.D., used at 225°C, injection port 250°C. (Laboratory relative polarity value⁴ + 100 to "standard" SP2250).

Mobile phase: nitrogen at about 40 ml min⁻¹ which had passed through a molecular sieve 13X to remove oxygen.

Recorder/integrator: Hewlett-Packard 3380A with input 10 mV or 1 V, at an attenuation setting 8 or 1024, respectively; using integration delay of 1 or 2 min for columns i or ii, respectively. An external standard method was set up and edited as necessary.

Nitromusks were kindly donated by Keith Harris & Co. (Australia). Toilettries studied were commercial supplies, some of which had been partly used, representing the normal consumer situation.

Procedure

A stock solution of about 80 mg per 100 ml each of musk ambrette and musk ketone in ethanol was prepared with vigorous shaking. This was serially diluted 1000-fold to provide the standard solution of about 80 µg per 100 ml each of the two nitromusks in ethanol. A 10-µl volume of this gave satisfactory peaks on the recorder at the coarsest range setting of the GCD attenuator. A similar solution of musk xylene and musk ketone was prepared.

Quantities of toilettries (1-10 µl) were diluted to 10 ml with ethanol, and 10-µl volumes assayed for musk ambrette and musk ketone by the external standard programme of the HP 3380A. Dilutions and assays were repeated and results on the two columns were obtained. Toilettries gave very minor peaks for constituents other than those corresponding to nitromusks, and these other peaks could be edited out of the integrator programme by rejecting peak areas less than a certain size (Fig. 1).

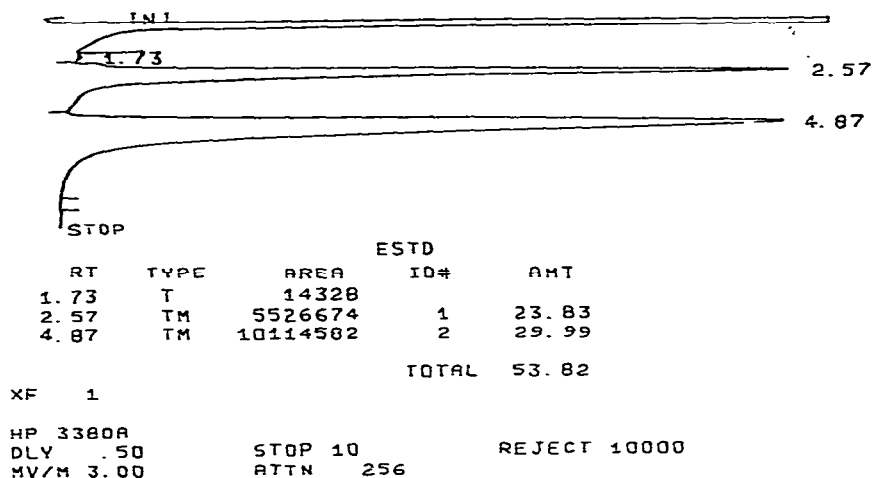
RESULTS AND DISCUSSION

The non-polar methylpolysiloxane column proved the better for assays, although the polar polyethyleneglycol column was better for distinguishing between musk ambrette and musk xylene.

The absolute retention times for musk ketone were under 5 min on column i, and 13-20 min on column ii. The relative retention times (musk ketone = 1.00) were: musk ambrette, column i 0.54 and column ii 0.49; musk xylene, column i 0.55 and column ii 0.45. (With equal mixtures of musk ambrette and musk xylene, on column a double peak was obtained.)

Integrator peak area response was similar for musk ketone and musk xylene on both columns, although the latter is a trinitro compound which might be expected to give greater response than the dinitro musk ketone. Unfortunately, musk ambrette yielded a less sensitive response, only about 66% of that for musk ketone on column i, and much less than this on column ii, which was operating at its maximum temperature. A better response to musk ambrette was achieved by lowering the operating temperature for column ii towards 200°C, but this gave an excessively long retention time for musk ketone.

Average results for several determinations on both columns are detailed in Table I. These values may not be in agreement with hard to obtain manufacturer's



LIST	ZRTW	ESTD
RT	ID#	AMT/AREA
2.66	1	000 004 312
4.89	2	000 002 965

XF 1. 1STD AMT 74.4

Fig. 1. Evaluation of a dilution of Cobb & Co. after-shave lotion (10 μ l in 10 ml alcohol). Musk ambrette is the peak, ID No. 1 with a retention time (RT) of 2.57 min (2.66 min with the external standard solution of 74.4 μ g per 100 ml —this shorter retention time is included within the $\pm 3\%$ retention time "window" set up in the programme). The "amount" (AMT) calculated by the programme needs to be doubled. Musk ketone is the peak ID No. 2. The absence of other peaks is noteworthy.

TABLE I

AVERAGE GAS CHROMATOGRAPHIC ASSAY RESULTS FOR NITROMUSKS IN TOILET-RIES

Note. The musk ambrette peaks may include some musk xylene owing to the closely matching retention times. However, double peaks were not observed on the polar column ii.

mg per 100 ml of:	Musk ambrette	Musk ketone	Musk xylene
Lotion "West" (Faberger)	1373	nil	nil?
Cologne "Moondrops" (Revlon)*	262	777	nil?
Splash-on "Brut 33"	261	nil	nil?
Lotion pre-electric shave (Aramis)	89	nil	nil?
Cologne "Tabac" (Maurer & Wirtz)	83	263	nil?
Lotion after-shave "Cobb & Co"	45	55	nil?
Splash-on "Cobb & Co." (Boots)	19	22	nil?
Cologne "Monsieur" (Lanvin)	8	20	nil?
Spray perfume "Tabu" (Lenthéric)*	nil	570	24

* Female toiletries.

formulae, but are the assay results from *used* toiletries. They indicate the extent of the potential problem, for a concentration of 250 mg per 100 ml of musk ambrette has caused a photoallergic response², as has a diluted cologne containing only 37.5 mg of this nitromusk per 100 ml. One male lotion has a concentration over five times higher than 250 mg per 100 ml, whilst a female cologne and a male splash-on (which might be used in liberal amounts) contain enough musk ambrette to present a potential hazard to some individuals. Shaving lotions, even though they may be apparently "safe", are applied to an exposed area of skin made sensitive by an abrading-scraping process and so may cause reaction.

No toiletry (over thirty examined) failed to show peaks indicative of nitromusks, although not all contained musk ambrette. A combination of nitromusks was commonly found.

REFERENCES

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